G-24-01

International Standard 5960

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Copper alloys — Determination of cadmium content — Flame atomic absorption spectrometric method

Alliages de cuivre — Dosage du cadmium — Méthode par spectrométrie d'absorption atomique dans la flamme

First edition — 1984-05-15

UDC 669.35 : 543.422 : 546.48 **Ref. No. ISO 5960-1984 (E)**

Descriptors : copper, copper alloys, chemical analysis, determination of content, cadmium, atomic absorption method.

Price based on 4 pages

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5960 was developed by Technical Committee ISO/TC 26, *Copper and copper alloys*, and was circulated to the member bodies in May 1982.

It has been approved by the member bodies of the following countries :

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Belgium	Iran	South Africa, Rep. of
Canada	Italy	Spain
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Australia

Printed in Switzerland

INTERNATIONAL STANDARD ISO 5960-1984 (E)

Copper alloys — Determination of cadmium content — Flame atomic absorption spectrometric method

1 Scope and field of application

This International Standard specifies a flame atomic absorption spectrometric method for the determination of the cadmium content of cadmium-containing copper alloys listed in International Standards.

The method is applicable to products having cadmium contents between 0,000 5 and 2,0 % (m/m).

2 Principle

Dissolution of a test portion in fluoroboric-nitric acid mixture. After appropriate dilution, aspiration of the test solution into an air-acetylene flame, and determination of the cadmium content by spectrometric measurement of the absorption of the 228,8 nm line emitted by a cadmium hollow-cathode lamp.

3 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

3.1 Fluoroboric-nitric acid, attack reagent.

Mix together 300 ml of boric acid, 40 g/l solution, 30 ml of hydrofluoric acid, 40 % (V/V) solution, 500 ml of nitric acid, ϱ 1,4 g/ml, and 150 ml of water.

3.2 Copper, 20 g/l base solution.

Transfer 20,0 g of cadmium-free copper to a 1 000 ml polytetrafluorethylene, polypropylene, or low-pressure polyethylene beaker. Add 800 ml of the attack reagent (3.1), warm until the copper is dissolved, then boil until nitrous fumes have been expelled. In the case of polyethylene or polypropylene beakers, use a water bath for heating. Allow to cool and transfer the solution to a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

50 ml of this solution contain 1 g of copper and 40 ml of the fluoroboric-nitric acid mixture (3.1).

3.3 Cadmium, standard solution corresponding to 1,000 g of Cd per litre.

Weigh, to the nearest 0,000 1 g, 1,000 g of cadmium metal (purity, $\geqslant$ 99,99 %). Transfer to a 250 ml beaker. Add 10 ml of water and 5 ml of nitric acid, ϱ 1,4 g/ml. Cover and warm gently (if necessary) until the cadmium metal is dissolved. Boil gently until nitrous fumes have been expelled, then allow to cool. Transfer the solution to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 1 mg of Cd.

3.4 Cadmium, standard solution corresponding to 0,050 g of Cd per litre.

Place 50,0 ml of the standard cadmium solution (3.3) in a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

1 ml of this standard solution contains 50 µg of Cd.

3.5 Cadmium, standard solution corresponding to 0,005 g of Cd per litre.

Place 100,0 ml of the standard cadmium solution (3.4) in a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

1 ml of this standard solution contains 5 µg of Cd.

4 Apparatus

Ordinary laboratory apparatus, and

4.1 Beakers, polytetrafluorethylene, polypropylene, or polyethylene, capacity 250 ml.

4.2 Burette, graduated in 0,05 ml.

4.3 Atomic absorption spectrometer, equipped with an air-acetylene burner.

4.4 Cadmium hollow-cathode lamp.

4.5 Compressed air supply.

4.6 Cylinder of acetylene.

5 Sampling[1)]

5.1 Test sample

The test sample shall be in the form of fine chips or drillings, obtained by milling or drilling, with a maximum thickness of 0,5 mm.

6 Procedure

6.1 Test portion

Weigh, to the nearest 0,000 1 g, about 1 g of the test sample (5.1).

6.2 Preparation of calibration graph

Prepare a calibration graph each time a series of samples is analysed.

6.2.1 Preparation of standard matching solutions

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of the standard cadmium solution (3.4 or 3.5), and copper base solution (3.2) as shown in tables 1, 2 or 3, depending on the expected cadmium content. Dilute to the mark and mix.

Table 1 — Cadmium content between 0,000 5 and 0,1 % (*m/m*)

Standard cadmium solution (3.5) ml	Standard cadmium solution (3.4) ml	Mass of cadmium contained mg	Copper base solution (3.2) ml	Mass of copper contained g	Corresponding cadmium content of sample % (*m/m*)
0*	—	0	50	1	0
1	—	0,005	50	1	0,000 5
2	—	0,01	50	1	0,001
6	—	0,03	50	1	0,003
10	—	0,05	50	1	0,005
15	—	0,075	50	1	0,007 5
20	—	0,10	50	1	0,010
—	6	0,30	50	1	0,030
—	10	0,5	50	1	0,050
—	15	0,75	50	1	0,075
—	20	1,0	50	1	0,100

* Blank test on reagents for calibration graph.

Table 2 — Cadmium content between 0,05 and 1,0 % (*m/m*)

Standard cadmium solution (3.4) ml	Mass of cadmium contained mg	Copper base solution (3.2) ml	Mass of copper contained g	Corresponding cadmium content of sample % (*m/m*)
0*	0	5	0,1	0
1	0,05	5	0,1	0,05
2	0,10	5	0,1	0,1
6	0,3	5	0,1	0,3
10	0,5	5	0,1	0,5
15	0,75	5	0,1	0,75
20	1,0	5	0,1	1,0

* Blank test on reagents for calibration graph.

1) An International Standard dealing with the sampling of copper alloys is in preparation.

Table 3 — Cadmium content between 0,5 and 2,0 % (m/m)

Standard cadmium solution (3.4) ml	Mass of cadmium contained mg	Copper base solution (3.2) ml	Mass of copper contained g	Corresponding cadmium content of sample % (m/m)
0*	0	2,5	0,05	0
5	0,25	2,5	0,05	0,5
10	0,5	2,5	0,05	1,0
15	0,75	2,5	0,05	1,5
20	1,0	2,5	0,05	2,0

* Blank test on reagents for calibration graph.

NOTES

1 The range of standard matching solutions is appropriate for most current models of equipment of average performance. The range and operating conditions should be selected for optimum measurements by the particular equipment available.

2 The presence of copper and attack acid in the standard matching solutions compensates for chemical interaction effects of these species.

6.2.2 Adjustment of the apparatus

Fit the cadmium hollow-cathode lamp (4.4) into the apparatus (4.3), switch on the current and allow to stabilize. Adjust the current, the sensitivity and the aperture of the slit according to the characteristics of the apparatus. Adjust the wavelength in the region of 228,8 nm to minimum absorbance. Adjust the pressure of the air and acetylene according to the characteristics of the aspirator burner.

6.2.3 Spectrometric measurements

Aspirate the appropriate series of standard matching solutions (6.2.1) in succession into the flame and measure the absorbance of each. Take care to maintain a constant rate of aspiration throughout the preparation of the calibration graph. Spray water through the burner after each measurement.

6.2.4 Plotting the calibration graph

Plot a graph having, for example, the masses, in milligrams, of cadmium contained in 100 ml of the standard matching solutions as abscissae, and the corresponding values of the measured absorbances, reduced by the value of the absorbance measured in the blank test on reagents for calibration graph (6.2.1, term 0), as ordinates.

6.3 Determination

6.3.1 Preparation of the test solution

Transfer the test portion (6.1) to a 250 ml PTFE, polypropylene or low pressure polyethylene beaker (4.1). Add 40 ml of the attack reagent (3.1). Cover with a watch glass and warm gently until the sample is dissolved, then heat at a temperature of nearly 90 °C until nitrous fumes have been expelled. Wash down the cover and sides of the beaker and allow to cool.

6.3.1.1 Cadmium content between 0,000 5 and 0,1 % (m/m)

Transfer the test solution (6.3.1) to a 100 ml one-mark, volumetric flask, dilute to the mark and mix.

6.3.1.2 Cadmium contents between 0,05 and 1 % (m/m)

Transfer the test solution (6.3.1) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

6.3.1.3 Cadmium contents between 0,5 and 2 % (m/m)

Transfer the test solution (6.3.1) to a 2 000 ml one-mark volumetric flask, dilute to the mark and mix.

6.3.2 Spectrometric measurements

6.3.2.1 Preliminary measurement

Carry out a preliminary measurement on the test solution (6.3.1) following the procedure specified in 6.2.3 at the same time as the spectrometric measurements are carried out on the standard matching solutions (6.2.1).

From the calibration graph (6.2.4), calculate the approximate concentration of cadmium in 100 ml of the test solution (6.3.1).

6.3.2.2 Bracketing measurements

Carry out a second measurement on the test solution (6.3.1) following the procedure specified in 6.2.3, by bracketing between two standard matching solutions of composition similar to that of the standard matching solutions (6.2.1), but having cadmium contents which differ by smaller increments.

To prepare these standard matching solutions, follow the procedure specified in 6.2.1, using, however, suitable quantities of standard cadmium solutions (3.4 or 3.5).

6.4 Blank test

Carry out a blank test at the same time as the determination and following the same procedure, using the same quantities of reagents and of pure copper as for the determination but omitting the test portion.

6.5 Check test

Make a preliminary check of the apparatus by preparing a solution of standard material or a synthetic sample containing a known amount of cadmium and of composition similar to the material to be analysed, and carrying out the procedure as specified in 6.1 to 6.4.

7 Expression of results

7.1 Concentration of the test solution

The cadmium concentration, c, expressed in milligrams of cadmium per 100 ml of test solution, is given by the formula

$$c = c_1 + (c_2 - c_1)\frac{A_0 - A_1}{A_2 - A_1}$$

where

c_1 is the concentration, in milligrams of cadmium per 100 ml, of the standard matching solution of lower concentration, used for the bracketing measurement (6.3.2.2);

c_2 is the concentration, in milligrams of cadmium per 100 ml, of the standard matching solution of higher concentration, used for the bracketing measurement (6.3.2.2);

A_0 is the value of the absorbance corresponding to the concentration of the test solution (6.3.1);

A_1 is the value of the absorbance corresponding to concentration c_1;

A_2 is the value of the absorbance corresponding to concentration c_2.

7.2 Cadmium content of the sample

The cadmium content of the sample, expressed as a percentage by mass, is given by the formula

$$\frac{(c - c_3)}{10\,m} \times r_D$$

where

c is the concentration, expressed in milligrams of cadmium per 100 ml, of the test solution (6.3.1), calculated in accordance with 7.1;

c_3 is the concentration, expressed in milligrams of cadmium per 100 ml, of the blank test solution (6.4);

m is the mass, in grams, of the test portion (6.1);

r_D is the ratio between the volume of the test solution (6.3.1) and the standard matching solutions (6.2.1).

8 Test report

The test report shall include the following particulars :

a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard or regarded as optional.